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Abstract

A reduced activation modification of the ferritic martensitic steel HT-9, called LIFE12, is being developed as a potential chamber wall material for Laser Inertial Fusion Energy (LIFE). As welding is an inevitable fabrication procedure for fusion components, a variety of conventional and solid-state welding processes were recently studied using this new material.

Electron beam (EB), tungsten inert gas (TIG), and laser welding were all performed to join the steel, followed by normalizing heat treatments between 750 °C and 1050 °C. From these possible heat treatments, a normalization scheme of 950 °C for one hour was chosen, given that this anneal caused minimal grain growth while still converging the hardness of the base metal with that of the fusion and heat-affected zones. Additionally, steel/steel diffusion bonds were developed using hot isostatic pressing (HIP) at temperatures between 850 °C and 1150 °C for two hours at 103 MPa. Interface tensile strength was over 600 MPa for HIP temperatures of 950 °C or higher. Additionally, no increased hardness nor voids were found at the diffusion bonded interface.

Keywords: RAFM steel, diffusion bonding, welding, first wall

1. Introduction

Ferritic martensitic (FM) steels have long been considered candidate structural materials for fusion power plants for their favorable resistance to swelling under irradiation [1, 2, 3]. Initial attempts in the 1980's to balance fracture toughness with creep strength resulted in 11-12Cr steels like MANET[4]. However, work was later done the fusion community to develop reduced activation ferritic martensitic steels that activate to lower levels under fusion irradiation than their standard ferritic martensitic counterparts [5]. The result was development of FM steels with 9% chromium content, such as the Japanese heat F82H and the European heat Eurofer-97.

However, some of these 9Cr steels have shown a sharp shift in the ductile to brittle transition temperature when irradiated at temperatures above 400 °C. FM steels with 12% chromium content, on the other hand, have shown far smaller ductile-to-brittle transition shifts when irradiated in temperature ranges between 400 °C and 600 °C [1]. Additionally, 12Cr steels have shown good creep rupture strength at these temperatures [6]. Since this is a very attractive operational temperature range for inertial confinement fusion blankets, the LIFE team is working with 12Cr FM steels as a starting point for materials design.

Since there are no reduced activation 12Cr ferritic martensitic steels currently being considered for fusion applications, it will be necessary to develop one. Capitalizing on considerable research done by the fission community, the LIFE team decided to modify the pre-existing FM steel Sandvik HT-9 into a reduced activation material. This was mainly done by removing molybdenum and nickel and substituting them with amounts of tungsten to make up solution strengthening and manganese to improve hardenability and ductility [1]. The team is calling this new material 'LIFE12.'

According to the Schaffler diagram, the changes to the alloying constituents drive down the nickel equivalent and chromium equivalent contents to destabilize austenite and ferrite enough to change its phase. Instead of a ferritic+martensitic+austenitic dominated phase, LIFE12 is a purely ferritic+martensitic phase, as opposed to F82H and Eurofer, which are only ferritic-martensitic after annealing. Because this difference places the material into a different space, it is important to re-characterize every aspect of the new material. In addition, techniques to join the material must also be developed and characterized. And while considerable effort has been spent characterizing HT-9 joints [7], the LIFE12 microstructure is sufficiently different such that different phases may develop, significantly changing the joint character. To this end, we bonded samples of LIFE12 together using a variety of weld processes, including electron-beam welding, tungsten inert gas welding, laser welding, and diffusion bonding.

2. Approach

Heats of LIFE12 were produced via vacuum induction melting and were hot-rolled at 1100 °C into 5 mm thick sheets. Though the material is still early in development and future melts will improve upon this heat, modifications are expected to cause minimal microstructural changes, making this a suitable time to learn some of the characteristics of joining processes.

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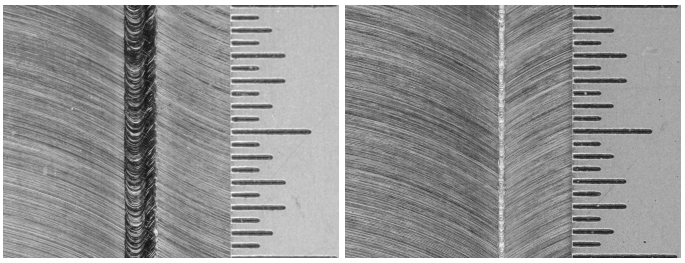
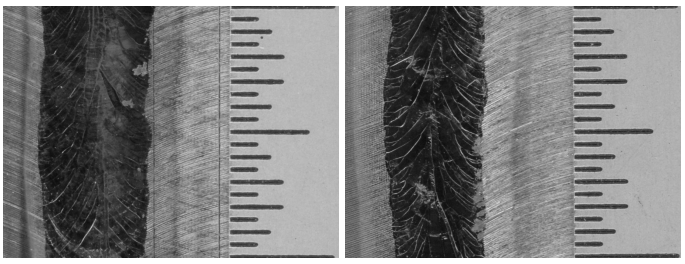
2.1. Welding processes

To imitate the expected thin-walled geometry of the LIFE first walls, samples for welding were ground to 1 mm thickness and cut into 152.4 mm [6”] long × 19 mm [0.75”] wide segments, which were then welded along their lengths using electron beam, tungsten inert gas, or laser welding.

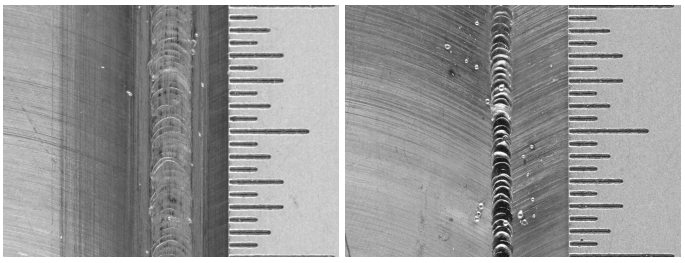
Electron beam welds were produced with a beam travel speed of 762 mm [30”] per minute at 80 K.V., a beam current of 4.5 mA, and a filament current of 5.3 mA. The beam shape was a DC spot with a gun to work distance of 9mm. This achieved 100% penetration.



(a) Electron beam weld



(b) Tunsten inert gas weld



(c) Laser weld

Figure 1: Front (left) and back (right) of three different weld beads. Tick marks are 0.5 mm.

Tungsten inert gas welds were performed using two techniques: with a filler rod and without a filler rod. In both cases, a Miller syncowave 250 was used with a DC straight electrode. A copper holding fixture was used to hold plates together. For the TIG weld with a filler rod, the ends of the plates were tacked with a gap between plates of 1.27 mm [0.050”]. A 2.54 mm [0.1”] diameter rod was used as filler. Using 35 CFH argon back purge gas and 15 CFH argon welding gas with a travel speed of 152.4 mm [6”] per minute, we achieved a 100% penetration weld. The same conditions were used for the weld without rod, except there was no gap between plates prior to welding.

After welding, various normalization temperatures were attempted between 750 °C and 1150 °C, all in an inert argon environment for one hour. In each case, the sample was rapidly heated and water-quenched.

Following annealing, micrography and electron backscatter scanning diffractometry were performed and nano- and microhardness measurements were taken. In addition, bending tests were conducted on small bending bars (38.1 *mm* [1.5″] long × 2.54 mm [0.1″] wide × 1 mm [0.039″] thick) to gauge the relative flexure capabilities of the different weldments.

2.2. Diffusion bonding methodology

Concurrent to the welding study, we also developed the necessary methodology to diffusion bond samples of LIFE12 using hot isostatic pressing (HIP). This process is achieved by bringing the volume to high temperature and applying very high pressurized gas around a vacuum tight shroud material which deforms to apply isostatic pressure on the work parts. To accomplish this, we encapsulated two LIFE12 samples in a thin-walled (1.27 mm) stainless steel can (see Fig 2). The can itself was made vacuum tight via electron beam welding after being evacuated.

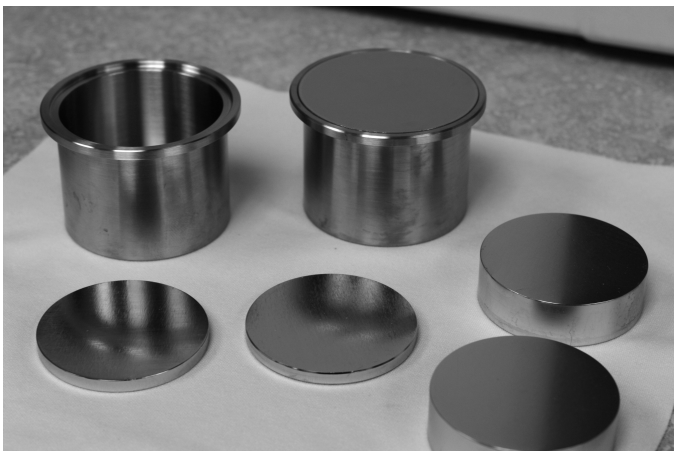


Figure 2: Contents of the HIP can, clockwise from upper left: empty HIP can, packed HIP can, two stainless steel spacer disks, two LIFE12 steel disks.

Because diffusion bonding works via atomic migration across an interface, bonding surfaces must be extremely clean and smooth prior to attempting the bond. This makes surface preparation perhaps the most critical step of the diffusion bonding process. To get the surface as clean and smooth as possible, we therefore followed the procedure outlined below:

1. Visual inspection for damage and cleanliness
2. High Pressure Clean and Rinse w/ Brulin 1990GD detergent and processed water
3. Ultrasonic cleaning and rinse of parts in 3-10% Brulin 815GD detergent in processed water at 43 – 54 °C.
4. Dry parts w/ filtered shop air/nitrogen
5. Passivate per ASTM A967, reference section 5.8
6. Perform free iron test reference MEL99-009-OK

7. Ultrasonic cleaning and rinse of parts in DI water
8. Dry parts w/ filtered shop air/nitrogen

After cleaning, the parts were baked out in an oven at 110 °C for one hour to ensure dryness and were immediately enclosed in a stainless steel can via e-beam welding. The sealed cans were heated in a HIP furnace at temperatures 850 °C, 950 °C, 1050 °C and 1150 °C for two hours at 103 MPa. The rate of heating and cooling was 450 °C per hour.

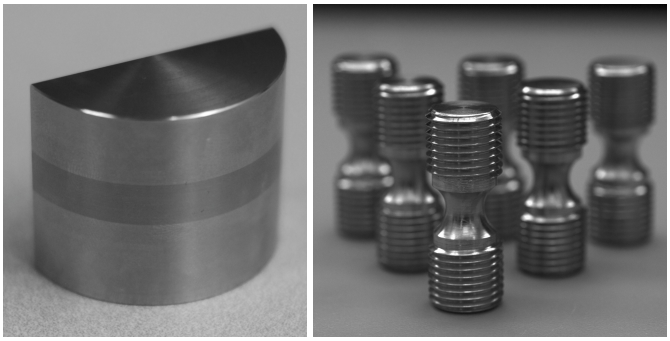


Figure 3: Bonded metal after removal of HIP shroud (left). Darker color at center is LIFE12, while the lighter color is stainless steel 316. From this, six tensile bars were extracted (right).

Because the available FM steel material was only 5 mm thick, and we desired to make tensile test bars from the bonded product, stainless steel 316 substrates were also bonded to the LIFE12 material to increase thickness. The resulting cylinder, after bonding, was 48.25 mm [1.9"] diameter by 38.1 mm [1.5"] tall. From this cylinder, six 12.7 mm [0.5"] diameter cylindrical tensile bars were cut for strength measurement of the diffusion bond (see Fig. 3).

Each tensile cylinder was pulled to failure under quasi-static loading at room temperature in an air environment. Further analysis was completed via microscopy and electron backscatter diffractometry. Additionally, nanohardness tests were conducted across the diffusion bonded section to check for hardness variation at the bond.

3. Results

LIFE12 appears to be an easily weldable steel, as all methods produced good quality bonds, with no noticeable stress cracking or porosity. Additionally, we identified an annealing recipe that normalizes the microstructure to improve overall structural integrity.

3.1. Welding

A cross section of the electron beam weld revealed a fusion zone consisting of almost entirely ferrite grains that were 50 – 200 μm in diameter (see Fig. 4a). Martensite was only present sporadically between large ferrite grains.

The fusion zone of the TIG weld is far larger than in either the e-beam or laser weld (see Fig. 4b). In this zone, there is considerably more martensite grains present than in the e-beam weld. The ferrite grains are very large in proportion to the weld cross-section, measuring nearly 1 mm long in some cases. The heat affected zone appears to be at least 300 μm wide, with a clear line dileneating the large ferrite grains of the fusion zone with much smaller ferrite grains (40 μm) interspersed with a lot of martensite.

Finally, the laser weld fusion zone appeared similar in dimension to the e-beam weld, but contained far more martensite grains (see Fig. 4c). Additionally, the ferrite grains are smaller than the e-beam weld (approximately 100 μm).

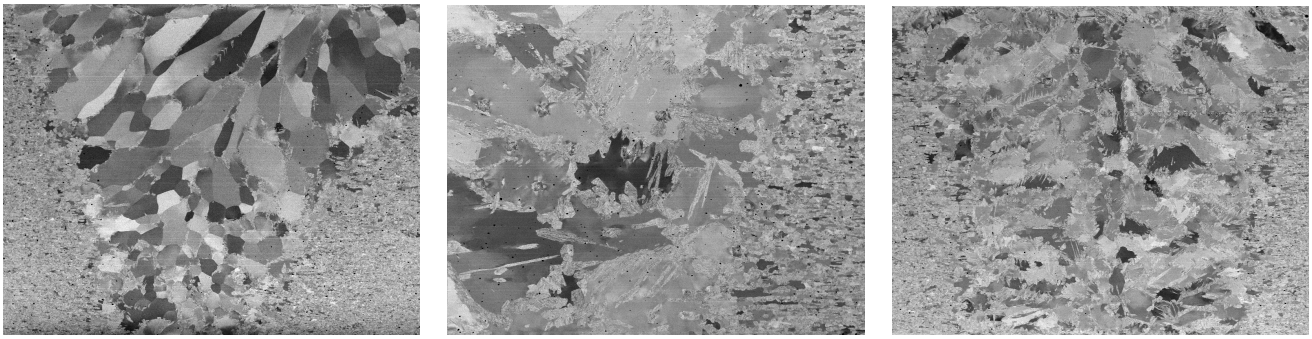
For reference, the base metal grain size is only 5 μm, and is roughly 77% martensite and 23% ferrite (estimated by grain area in EBSD) arrayed in semi-organized bands through the material.

Three factors have influenced the microstructure of the three weld techniques: temperature reached, time at temperature, and heating/cooling rate. The e-beam weld appears to have cooled slowly enough to transform austenite grains into ferrite, forcing only a few martensite grains. In contrast, the TIG weld maintained an austenisting temperature for much longer, allowing larger prior-austenite grains that, through relatively fast cooling, transformed into both ferrite and martensite. The laser weld appeared to have a similar cooling rate to the TIG weld based on the amount of ferrite and martensite, but remained at austenizing temperatures for less time, resulting in smaller prior-austenite grain sizes.

Prior to annealing, the fusion zone was considerably softer than the base metal and heat-affected zone (a hardness of 2.7 GPa instead of 3.7 GPa), due to its lack of martensite. However, annealing the material effectively re-introduced martensite into the weld and normalized this difference. Anneals for one hour at either 750 °C or 850 °C had the effect of only increasing grain size without significantly affecting the ratio of martensite to ferrite (Fig. 5b). However, anneals at 950 °C and 1050 °C cooled to reveal a dominant martensitic structure throughout the fusion zone, heat-affected zone, and base material (Figs. 5c & 5d). Both 950 °C and 1050 °C anneals yielded about 77% martensite, the same ratio as the base metal. This indicates the austenitization temperature occurs between 850 °C and 950 °C.

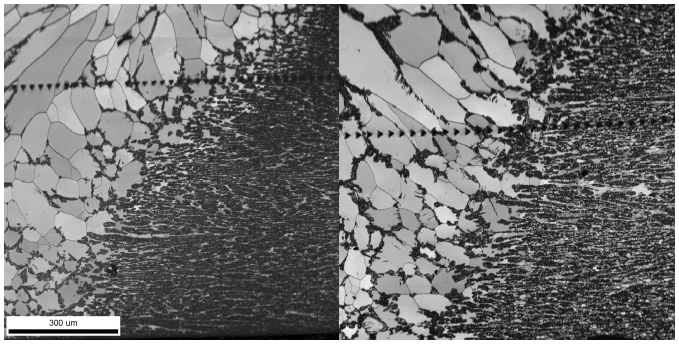
In accordance with the grain growth of annealing, the hardness in the fusion zone, heat-affected zone and base metal all dropped significantly during lowest temperature anneal (see Fig. 7 for example of e-beam weld response to annealing). However, after the 850 °C anneal and more so after the 950 °C anneal, hardness was regained due to the increase in martensite. By the 950 °C, the fusion zone was nearly as hard as the heat-affected zone and base metal, effectively erasing the previous discontinuity of the weld. Higher temperature anneals were therefore unnecessary, and only further increased the grain size of the material. Prior to annealing the base metal had an average grain diameter of approximately 5 μm, which increased up to a maximum of 10.5 μm after the 1050 °C anneal (see Fig. 6).

Small bending specimens served to give qualitative comparisons of the relative strength of each weld to the base metal strength. The size and scope of the work did not allow for comprehensive quantitative strength data at this time. For reference, the base material was measured to have a tensile yield strength

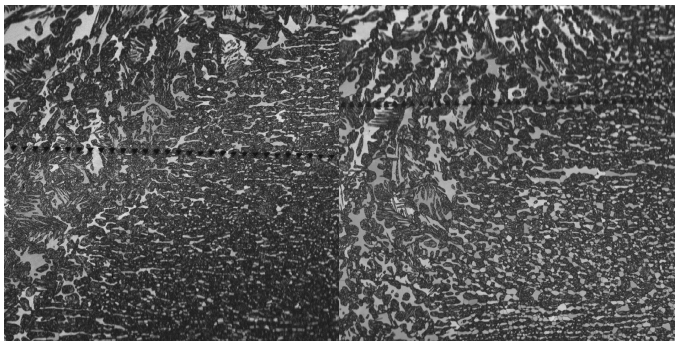


(a) Electron beam weld (b) TIG weld (c) Laser weld

Figure 4: Backscatter images of cross sections of the 100% penetration welds across a 1 mm thick LIFE12 sheet.



(a) EB as-welded (b) 850 °C/ 1 hr



(c) 950 °C/ 1 hr (d) 1050 °C/ 1 hr

Figure 5: Annealed EBSD images of cross-sectioned electron-beam welds after various anneals. Light grains are ferrite, while dark grains are martensite. The 950 °C/ 1 hr anneal normalized the weld's martensite fraction to that of the base material.

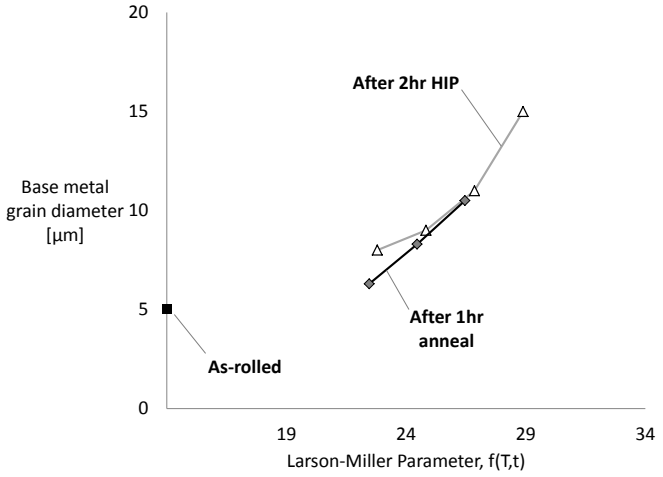


Figure 6: Grain size increased from 5 μm as-rolled up to 10.5 μm after the highest temperature anneal (1150 °C).

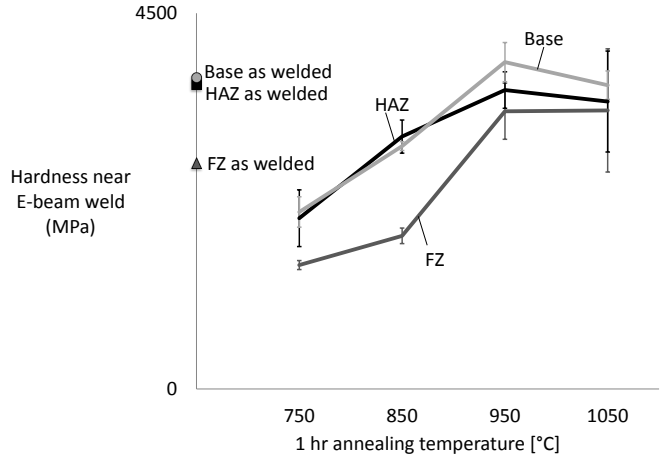


Figure 7: Hardness decreased from the as-welded condition due to grain growth, but recovered as the martensite percentage increased. The three regions near the e-beam weld (fusion zone, heat-affected zone, and base metal) converged when annealed at or above 950 °C. TIG welds and laser welds responded similarly.

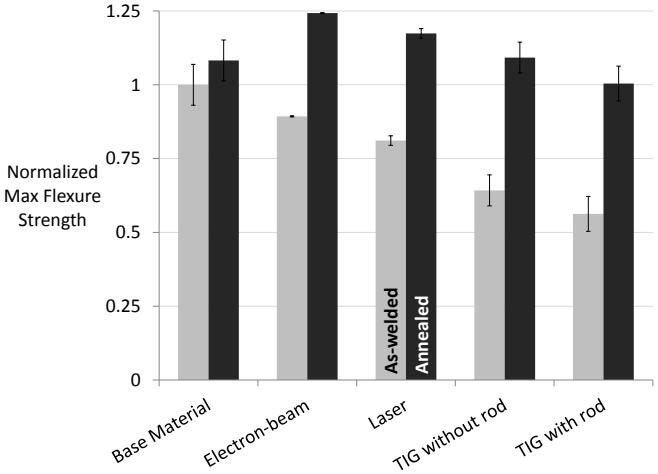


Figure 8: The bending strength of the as-welded joints are all considerably weaker than the base metal, but annealing recovers their strength to as good or better than the base metal.

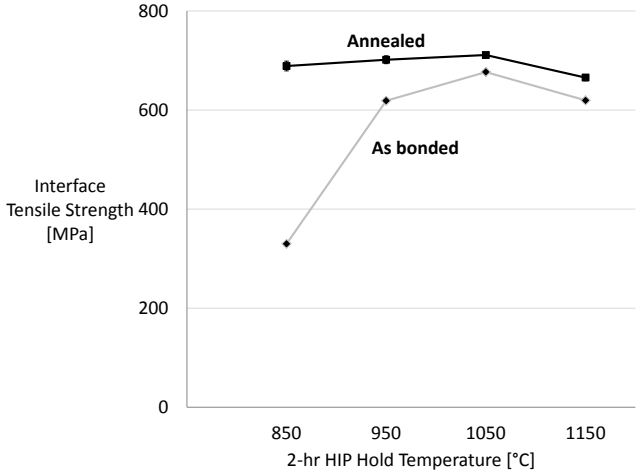


Figure 9: The tensile strength of the diffusion bond interface was similar when bonded at (950 °C/2 hr) or higher. Interestingly, the weakest bond, joined at 850 °C, improved strength with a (950 $°C/1$ hr) anneal.

of 727 MPa and an ultimate tensile strength of 931 MPa.

For every weld technique, the maximum flexure strength of the as-welded condition was less than the base metal strength. The strongest of these was the e-beam weld, then the laser weld, followed by the TIG weld with and without a filler rod. As predicted, annealing the sample for one hour at 950 °C improved the structural quality of all welds. After annealing, the same order of strengths between welds was retained, but all welds were as strong or stronger than the original base metal.

3.2. Diffusion Bonding

HIP bonds conducted at 950 °C and above produced similar tensile strengths of approximately 600 MPa or higher: at least 75% of the ultimate tensile strength of the base material. Below this bonding temperature, however, the bond attempt failed with a strength of only 330 MPa. Interestingly, when annealed at 950 °C for one hour, the 850 °C bond was able to regain the strength of the other bonds (over 600 MPa).

Since the base metal microstructural is invariably affected by diffusion bonding, it is desirable to minimize the bonding temperature while also creating a strong interfacial bond. For this system, a HIP bond of 950 °C for two hours appears to be the best option for fabrication. Using this procedure, the base metal grain is increased to approximately 9 μm in diameter.

Visual inspection showed no obvious joint line or porosity after any of the bond temperatures (interface was only visible as a slight orientation difference via EBSD). Nor was there any evidence of increased hardness near the interface, as investigated via nano-hardness testing. Additionally, the ferrite-martensite ratio was maintained at 23%/77%, just as it was prior to diffusion bonding (and after a one hour anneal at 950 °C).

4. Conclusions

Despite differences in this particular steel's microstructure from other reduced activation ferrite martensitic steels, all attempted joining methods were successful. The steel is easily weldable, not cracking in the way of some martensitic steels upon cooling.

Annealing the samples at 950 °C for one hour sufficiently normalizes the fusion and heat-affected zones to match grain size and phase ratio well with the base material. The austenitic transition temperature was found to be somewhere between 850 °C and 950 °C.

Diffusion bonding was also implemented successfully using the outlined methodology. A HIP cycle of 950 °C for two hours at 103 MPa was sufficient to produce a bond strength nearing the strength of the base material. At this temperature, the effects on the base metal microstructure were minimal.

Acknowledgments

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